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Solar Energy Systems - Standards for Rubber Hose Used With Liquids Above Their Boiling Points

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
National Engineering Laboratory
Center for Building Technology
Structures and Materials Division
Washington, DC 20234

July 1981

Final Report

Issued September 1981

Prepared for

Department of Energy

Office of Solar Heat Technologies
Conservation and Renewable Energy
Washington, DC 20585

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SOLAR ENERGY SYSTEMS - STANDARDS FOR RUBBER HOSE USED WITH LIQUIDS ABOVE THEIR BOILING POINTS

Robert D. Stiehler

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National Bureau of Standards
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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, Secretary
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director

ABSTRACT

Class AT hose in ASTM Standard D 3952-80, Specification for rubber hose used in solar energy systems, is specified for use with aqueous liquids above 100°C. The lining of this hose is subjected to immersion tests at 100°C. The purpose of this study is to determine whether immersion tests in aqueous liquids above maximum service temperature are necessary in the hose standard.

The results of an interlaboratory test indicate that Class AT hose should be subjected to immersion tests above maximum service temperature. By inference, Class N hose used with a volatile heat transfer liquid at a temperature above its boiling point should be similarly tested above maximum service temperature.

The study also indicates that ASTM Standard Reference Coolant (ethylene glycol base) is not stable at 150°C. In addition to the hose lining, the stability of the heat transfer liquid above maximum service temperature must be determined to assure satisfactory performance of the system.

A proposed revision of ASTM D 3952 is included in the report. It provides for immersion tests above maximum service temperature of Class AT and Class N hose linings used with heat transfer liquids above their boiling points.

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1. INTRODUCTION

1.1 BACKGROUND

NBSIR 79-1917, Solar Energy Systems - Standards for Rubber Hose [1]¹, reported on a study of rubber hoses used to convey heat transfer liquids below their boiling points. A proposed standard for rubber hose used in solar energy systems was included in the report. This standard with minor modifications has been adopted by the American Society for Testing and Materials (ASTM) under the designation D 3952-80 [2]. This standard covered any rubber hose used in solar energy systems. When this standard was balloted in ASTM Committee D-11 on Rubber and Rubber-Like Materials, a negative vote was received since the standard did not provide for testing hoses above the boiling points of the heat transfer liquids. Class AT hose is specified for use with aqueous liquids at temperatures above the boiling point of water. This negative was withdrawn provided further studies were made to test hose linings immersed in aqueous liquids above their boiling points, and appropriate revision of the standard was made based on the results of the study.

1.2 OBJECTIVES

This study had the following objectives:

1. To conduct an interlaboratory test on typical commercial hose linings immersed in aqueous antifreeze solution at 100 and 150°C,
2. To determine whether tests at temperatures above the boiling point of the liquid are necessary in the standard, and
3. To propose a revision of the standard, if necessary, to assure satisfactory performance of hose under all conditions in solar energy systems.

¹ The numbers in brackets refer to the references in section 8 of this report.

2. PROBLEM ASSESSMENT

2.1 TEST METHOD

In order to test hose linings in liquids at temperatures above their boiling points, a test method is needed since ASTM D 471 [3] provides only for testing at temperatures below the boiling point of liquids. A pressure vessel is required for tests above the boiling points.

2.2 PRESSURE VESSEL

ASTM D 454 [4] and D 572 [5] require pressure vessels to test rubber in air or oxygen under pressure. These vessels can be used to test rubber in liquids under pressure provided the metal used to construct these vessels does not react with the liquid. These vessels are expensive and not available in many laboratories. An alternative is to design and construct a pressure vessel which accomodates the glass test tubes specified in ASTM D 471. Thus, tests at temperatures below and above the boiling of the liquid can be made in the same glass containers.

3. LABORATORY STUDY

3.1 APPARATUS

A pressure vessel was designed using commercial pipe closed on one end with a welded plate and on the other end with a welded flange and blank end fitting. The blank end fitting was drilled and tapped to accommodate a pressure relief valve opening at about 1000 kPa. The vessel shown in figure 1 was designed to hold seven test tubes 38 mm in outside diameter and about 300 mm long. A commercial pipe gasket was used to effect a seal between the flange and end blank.

3.2 MATERIALS

Six commercial hose lining vulcanizates (two each from three hose manufacturers) were supplied in the form of sheets about 2 mm in thickness, 150 mm long, and either 100 or 150 mm in width. Vulcanizates 1 and 2 were based on silicone rubber and vulcanizates 3, 4, 5, and 6 were based on EPDM rubber.

The immersion liquid was ASTM Reference Coolant conforming to ASTM D 3585 [7], diluted with an equal volume of distilled water.

3.3 SPECIMENS

Fifteen Type C specimens of each hose lining vulcanizate were cut from the sheets allocated to each laboratory participating in the interlaboratory test. Three specimens were selected at random for each of the following treatments:

1. none
2. immersion in test liquid for 166 hours at 100°C
3. immersion in test liquid for 332 hours at 100°C
4. immersion in test liquid for 166 hours at 150°C
5. immersion in test liquid for 332 hours at 150°C

3.4 PROCEDURE

3.4.1 Tests at 100°C

Specimens immersed in test liquid at 100°C were tested in accordance with ASTM D 471. The immersion tests for 332 h were started one week before those at 166 h, so that the properties of all specimens could be measured together after immersion.

3.4.2 Tests at 150°C

Specimens were placed in glass test tubes containing 150 cm³ of test liquid as described in ASTM D 471. The three specimens of each vulcanizate were placed in a separate tube. The six test tubes were placed in the pressure vessel described in 3.1. About 100 cm³ of test liquid was added to the vessel outside the tubes to facilitate heat transfer. The vessel was closed with the blank end fitting using a commercial pipe gasket to effect a tight seal between

flange and blank end fitting. The vessel was then heated in a laboratory oven at 150°C for the prescribed time. After heating the vessel was allowed to cool to room temperature before opening it. The condition of the liquid and specimens was examined visually after the prescribed immersion periods and prior to the measurements described in 3.5. The immersion tests for 332 h were started one week before those for 166 h so that the properties of all specimens could be measured together, except by laboratory 3 which had only one pressure vessel. In this laboratory, measurements were made after each immersion test was completed.

3.5 MEASUREMENTS

3.5.1 Change in Mass

The mass of each specimen was determined before and after immersion in the test liquid for the prescribed periods at the prescribed temperatures in accordance with ASTM D 471. The change in mass was calculated in percent of the original mass.

3.5.2 Tensile Properties

The tensile strength and ultimate elongation of the untreated and treated specimens were determined in accordance with ASTM D 412. The change in these properties due to immersion was determined in percent of the values for the untreated specimens.

3.6 RESULTS

The interlaboratory test described in Appendix I was planned to include five participating laboratories. Only three laboratories completed the tests. Two laboratories used pressure vessels described in 3.1; the other laboratory used conventional pressure vessels for testing rubber under oxygen pressure [5]. The remaining two laboratories did not return their data. The following results were obtained by the three laboratories completing the tests.

3.6.1 Change in Mass

Table 1 gives the results on percent increase in mass of the six vulcanizates for the four conditions of immersion in ASTM Reference Coolant solution. All values are less than 10 percent at 100°C. At 150°C, four values obtained by laboratory 3 for vulcanizates 3, 4, and 6 are slightly over 10 percent. Specimens immersed 166 and 332 h have essentially the same percentage increase in mass.

The higher temperature causes slightly greater increase in mass of EPDM vulcanizates (vulcanizates 3, 4, 5, and 6), but the increase at 150°C is not deemed sufficient to affect the use of the hose in solar energy systems. Changes in length of the dumbbell specimens measured at NBS confirm this conclusion.

3.6.2 Tensile Strength

Table 2 gives the original tensile strengths for the six vulcanizates and the percent change caused by immersion in the test liquid. The agreement among the original properties is good for vulcanizates 1, 2, 3 and 5. Laboratory 2 reports a lower value for vulcanizate 4 which causes all percent changes after immersion to be excessively high. Therefore, this value is probably anomalous and the mean of the other two laboratories is used to calculate the percent change after immersion. Laboratory 1 has a higher value for vulcanizate 6, but the percent changes after immersion indicate that the original value is less than 10 percent high.

The results for vulcanizates immersed in the test liquid at 100 and 150°C show no appreciable effect of temperature except for vulcanizate 2. At 100°C, the percent change in tensile strength of vulcanizate 2 is not significant, but at 150°C, the tensile strength decreases markedly and the percentage decrease becomes greater with time of immersion.

3.6.3 Ultimate Elongation

Table 3 gives the original ultimate elongation for the six vulcanizates and the percent change after the four conditions of immersion in the test liquid. The agreement among the laboratories is good for all vulcanizates except vulcanizate 4. The low value obtained by laboratory 2 for original elongation of vulcanizate 4 causes all percent changes after immersion to be positive instead of negative. Therefore, this value is anomalous and percent changes for this laboratory are based on the mean of the values obtained by the other two laboratories.

The results for vulcanizates 1, 3, and 4 show no appreciable effect of immersion in test liquid at either 100 or 150°C. The decrease in elongation of vulcanizate 2 is not appreciably different than that of the other vulcanizates at 100°C, but is definitely greater at 150°C and the decrease becomes greater with time of immersion. For vulcanizates 5 and 6, the decrease in elongation is greater at 100°C than at 150°C, but the time of immersion has no apparent effect; the cause of this behavior is not apparent.

3.6.4 Appearance of Specimens After Immersion

The physical appearance of the specimens after immersion was not appreciably changed other than a slight amount of swelling already noted in 3.6.1.

3.6.5 Appearance of Test Liquid After Immersion Tests

The mixture of ASTM Reference Coolant and water after the immersion tests at 100°C had a small amount of white sediment. The mixture in contact with vulcanizates 1, 2, 4, 5, and 6 had a dark reddish oily layer floating on the surface which may have been material extracted from the vulcanizates. The original green color of the mixture appeared unchanged.

After immersion tests at 150°C, the green color of the original mixture was no

colorless. The amount of sediment tended to be greater in tests at 150°C than in tests at 100°C and darker in color. The oily layer was not noticeable at 150°C but the mixture itself separated into two phases. The heavier phase was more viscous than the lighter phase and slightly colored.

In order to determine whether the observed appearance of the mixture after tests at 150°C was due to the rubbers or to temperature instability, the mixture was heated alone in the same manner as the immersion tests at this temperature. After heating, the green color of the mixture had disappeared, white sediment was present, and the mixture separated into two phases. The liquid phases and the sediment were lighter in color without the rubber and the amount of sediment appeared to be less, but quantitative measurements were not made. It was concluded that the rubber vulcanizates were not responsible for the observed changes in the test liquid but they may have accelerated the changes.

4. DISCUSSION

This study indicates that temperature and time of immersion are not critical for good vulcanizates. On the other hand, the results obtained with vulcanizate 2 indicate that tests at 100°C are misleading if hose made with this vulcanizate is used in solar energy systems at 150°C. Vulcanizate 2 conforms to the requirements in ASTM D 3952 and should be suitable for use in solar energy systems where the temperature of the heat transfer liquid is below 100°C. It is not satisfactory for use in systems where the temperature of the heat transfer liquid is frequently 150°C. Therefore, ASTM D 3952 needs revision so that hoses conforming to the standard are suitable for use at high temperatures.

The study also indicates that glycol-water mixtures may not be suitable for the heat transfer liquid in pressurized systems operating at high temperatures. Tests of the proposed heat transfer fluid are required to determine the stability of the liquid at the highest temperature expected in the system.

5. PROPOSED REVISION OF ASTM D 3952

The following revision of footnote B under Table 1 in ASTM D 3952 given in Appendix II is proposed to assure satisfactory performance of rubber hose used in solar energy systems with heat transfer liquids above their boiling points at atmospheric pressure:

-
- B The inner tube of Class A hose shall be immersed in a mixture containing equal volumes of ethylene glycol and water for 166 ± 2 h at $100 \pm 2^\circ\text{C}$. The inner tube of Class N hose shall be immersed in heat transfer liquid used in the solar energy system for 166 ± 2 h at a standard temperature listed in Recommended Practice D 1349 at least 20°C above the maximum service temperature, but not less than 100°C . The inner tube of Class AT and Class N hose used in a solar energy system at a temperature above the boiling point of the heat transfer liquid shall be immersed in the liquid contained in a pressure vessel (Note 5) for 166 ± 2 h at a standard temperature listed in Recommended Practice D 1349 at least 20°C above the maximum service temperature, but not less than 100°C .

CAUTION: The pressure chamber must have an air space equal to at least one-third its volume when it is closed for test at elevated temperature. The pressure vessel must be designed to withstand the vapor pressure of the liquid plus the pressure of the air at test temperature and must have a pressure release safety device. Allow the pressure vessel to cool to room temperature after the test before opening it.

NOTE 5: The pressure vessel may be one prescribed in Method D 454 and D 572 provided the heat transfer liquid does not react with the metal used to construct the vessel. Alternatively, a pressure vessel may be constructed from metal pipe and fittings. The pressure chamber is 305 mm high to accommodate the glass test tubes prescribed in Method D 471. A suggested construction is to weld a plate on one end and a flange on the other end of a nominal 125 mm (5 in) steel pipe. A blank fitting with a pressure release safety device and a gasket are used to seal the pressure chamber. This chamber accommodates seven glass test tubes 38 by 300 mm.

6. CONCLUSIONS

6.1 ASTM D 3952 is not adequate to assure satisfactory performance of rubber hose in solar energy systems operating with heat transfer liquids at temperatures above their boiling points at atmospheric pressure.

6.2 A revision of footnote B in table 1 of ASTM D 3952 can remedy the present deficiency in this standard.

6.3 Glycol-based antifreeze mixtures may not be stable at temperatures above their boiling points.

6.4 Both the hose lining and the heat transfer liquid must be tested at a temperature above the maximum service temperature to assure satisfactory performance in solar energy systems.

7. ACKNOWLEDGMENT

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Gates Rubber Company

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Leonard Walker, Underwriters' Laboratories
John Grimes, National Bureau of Standards
Chuck Knoll, Gates Rubber Company

The support of Active Heating and Cooling Division, Office of Solar Heat Technologies, the Department of Energy, sponsor of this project, is gratefully acknowledged.

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- [4] Standard test method for rubber deterioration by heat and air pressure. American Society for Testing Materials. D 454-53 (reapproved 1976); 1953 September. 4 p.
- [5] Standard test method for rubber deterioration by heat and oxygen pressure. American Society for Testing and Materials. D 572-73; 1973 May. 4 p.
- [6] Standard test methods for rubber properties in tension. American Society for Testing and Materials. D 412-80; 1980 July. 14 p.
- [7] Standard specification for ASTM Reference Coolant. American Society for Testing and Materials. D 3585-77; 1977 July. 5 p.

¹ Reference [1] can be obtained from the National Technical Information Service (NTIS), Springfield, VA 22161.

Reference [2] to [7] can be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

Table 1. Summary of Change-in-Mass Results¹

Vulcanizate:	1	2	3	4	5	6
166 h at 100°C:						
Laboratory 1	7.7	9.6	0.7	3.9	6.6	7.8
2	3.8	3.1	1.4	1.1	2.1	1.9
3	<u>3.4</u>	<u>2.6</u>	<u>1.2</u>	<u>1.7</u>	<u>2.1</u>	<u>2.2</u>
average	<u>5.0</u>	<u>5.4</u>	<u>1.1</u>	<u>2.2</u>	<u>3.6</u>	<u>4.0</u>
332 h at 100°C:						
Laboratory 1	1.6	4.3	0.1	6.8	1.1	2.0
2	3.5	3.9	1.9	1.6	2.7	2.9
3	<u>3.8</u>	<u>3.8</u>	<u>1.9</u>	<u>1.9</u>	<u>2.5</u>	<u>2.8</u>
average	<u>3.0</u>	<u>4.0</u>	<u>1.3</u>	<u>3.4</u>	<u>2.1</u>	<u>2.6</u>
166 h at 150°C:						
Laboratory 1	5.7	4.4	3.0	2.9	4.7	4.2
2	2.4	4.7	3.1	2.2	1.0	3.1
3	<u>4.8</u>	<u>4.6</u>	<u>9.5</u>	<u>7.2</u>	<u>6.5</u>	<u>12.1</u>
average	<u>4.3</u>	<u>4.6</u>	<u>5.2</u>	<u>4.1</u>	<u>4.1</u>	<u>6.5</u>
332 h at 150°C:						
Laboratory 1	5.9	5.1	3.0	2.4	5.2	4.6
2	4.5	6.9	7.9	2.0	4.9	4.4
3	<u>6.2</u>	<u>4.3</u>	<u>10.6</u>	<u>10.2</u>	<u>7.2</u>	<u>11.5</u>
average	<u>5.5</u>	<u>5.4</u>	<u>7.2</u>	<u>4.9</u>	<u>5.8</u>	<u>6.8</u>

¹ The numerical values represent the percent increase in mass after heating in ASTM Reference Coolant-solution.

Table 2. Summary of Tensile Strength Results

Vulcanizate:	1	2	3	4	5	6
Tensile Strength, MPa, of untreated specimens						
Laboratory 1	12.6	7.0	7.4	9.4	14.8	7.9
2	12.4	7.0	7.1	7.2 ^a	13.2	5.9
3	12.6	7.0	7.2	8.3	13.4	6.7
average	<u>12.5</u>	<u>7.0</u>	<u>7.2</u>	<u>8.9</u>	<u>13.8</u>	<u>6.9</u>
Percent change after heating in test liquid for 166 h @ 100°C						
Laboratory 1	-16.9	- 8.9	+ 6.5	+ 8.1	-10.7	+ 1.7
2	-12.3	- 7.9	+14.6	+22.4 ^b	+ 8.0	+11.8
3	- 1.6	-10.8	+ 6.7	+ 7.6	+ 6.7	+ 7.2
average	<u>-10.3</u>	<u>- 9.2</u>	<u>+ 9.3</u>	<u>+12.7</u>	<u>+ 1.8</u>	<u>+ 6.9</u>
Percent change after heating in test liquid for 332 h @ 100°C						
Laboratory 1	- 6.6	-11.9	+ 7.4	+12.5	+ 3.7	+ 7.0
2	- 2.2	-15.8	+18.4	+26.5 ^b	+ 8.0	+10.2
3	+ 1.6	-11.8	+15.4	+12.4	+13.4	+10.3
average	<u>- 2.4</u>	<u>-13.2</u>	<u>+13.7</u>	<u>+17.1</u>	<u>+ 8.4</u>	<u>+ 9.2</u>
Percent change after heating in test liquid for 116 h @ 150°C						
Laboratory 1	-10.4	-59.4	- 7.4	+22.1	-10.2	0.
2	-18.9	-32.7	- 5.8	+25.9 ^b	+ 1.5	+ 7.2
3	-23.5	-55.9	+ 1.0	+20.7	0.	+ 6.7
average	<u>-17.6</u>	<u>-49.3</u>	<u>- 4.1</u>	<u>+22.6</u>	<u>- 2.9</u>	<u>+ 4.6</u>
Percent change after heating in test liquid for 332 h @ 150°C						
Laboratory 1	-15.8	-73.3	- 8.3	+22.8	-14.4	-10.4
2	-16.8	-69.8	- 2.9	+10.7 ^b	- 9.5	- 5.9
3	-27.3	-70.6	- 2.9	+21.5	+ 5.2	0.
average	<u>-19.7</u>	<u>-71.2</u>	<u>- 4.7</u>	<u>+18.3</u>	<u>- 6.2</u>	<u>- 5.4</u>

^a This anomalous value is omitted in calculating the average.

^b These values are based on 8.86 MPa for the original value. The values based on 7.24 MPa are +49.5, +55.2, +54.3, and +35.2 percent, respectively.

Table 3. Summary of Ultimate Elongation Results

Vulcanizate:	1	2	3	4	5	6
Ultimate Elongation, percent, of untreated specimens						
Laboratory 1	650	360	390	590	530	240
2	640	340	380	380 ^a	510	210
3	670	350	370	560	490	230
average	<u>655</u>	<u>350</u>	<u>380</u>	<u>575</u>	<u>510</u>	<u>225</u>
Percent change after heating in test liquid for 166 h @ 100°C						
Laboratory 1	-12.3	-16.7	-17.9	-13.6	-32.1	-20.8
2	-10.9	-8.8	-10.5	-18.3 ^b	-19.6	-9.5
3	-9.0	-17.1	-5.4	-16.1	-18.4	-21.7
average	<u>-10.7</u>	<u>-14.2</u>	<u>-11.3</u>	<u>-16.0</u>	<u>-23.4</u>	<u>-15.1</u>
Percent change after heating in test liquid for 322 h @ 100°C						
Laboratory 1	-13.8	-19.4	-17.9	-22.0	-30.2	-20.8
2	-9.4	-20.6	-15.8	-16.5 ^b	-21.6	-19.0
3	-10.4	-20.0	-13.5	-5.4	-18.4	-21.7
average	<u>-12.2</u>	<u>-20.0</u>	<u>-15.7</u>	<u>-14.6</u>	<u>-23.4</u>	<u>-20.5</u>
Percent change after heating in test liquid for 166 h @ 150°C						
Laboratory 1	+4.6	-44.4	-15.4	-25.4	-11.3	-4.2
2	+1.6	-23.5	-5.2	-18.3 ^b	-9.8	0.
3	+4.5	-42.9	-5.4	-14.3	+2.0	+4.2
average	<u>+3.6</u>	<u>-36.9</u>	<u>-8.7</u>	<u>-19.3</u>	<u>-6.4</u>	<u>0</u>
Percent change after heating in test liquid for 332 h @ 150°C						
Laboratory 1	+7.7	-63.9	-12.8	-16.9	-9.4	0.
2	+7.8	-55.9	-10.5	-20.0 ^b	-13.7	0.
3	-3.0	-57.1	-13.5	-14.3	0.	0.
average	<u>+4.5</u>	<u>-59.0</u>	<u>-12.3</u>	<u>-17.1</u>	<u>-7.7</u>	<u>0.</u>

^a This anomalous value is omitted in calculating the average.

^b These values are based on 575 percent for the original value. The values based on 380 percent are +23.7, +26.3, +23.7, and +21.1 percent, respectively.

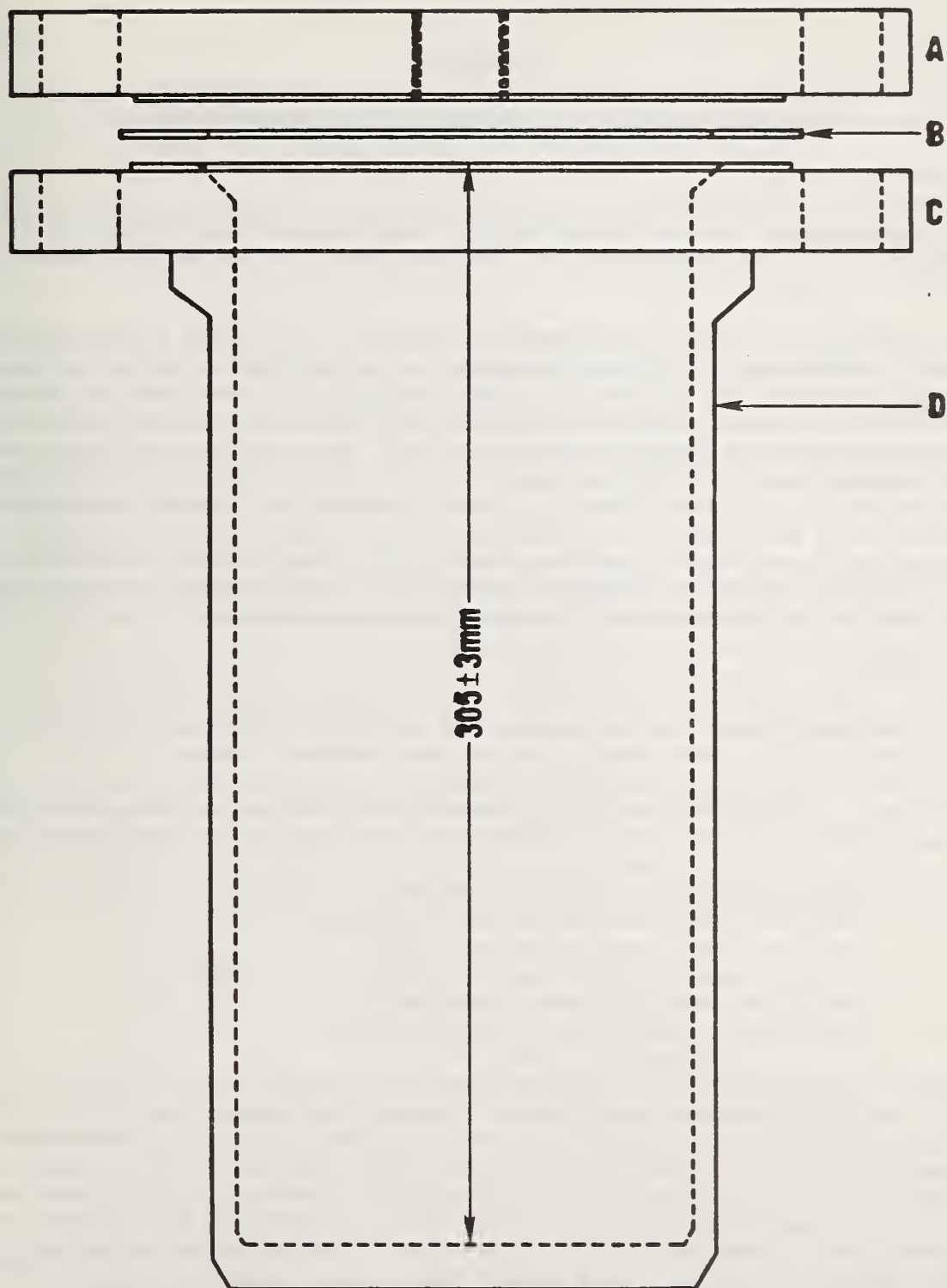


Figure 1. Pressure vessel

- A. Blank fitting drilled and tapped for pressure relief device.
- B. Gasket.
- C. Pipe flange fitting welded to pipe.
- D. Nominal 125 mm (5 in) pipe with plate welded to bottom end.

APPENDIX I

INTERLABORATORY TEST ON HOSE LINING VULCANIZATES

EQUIPMENT

Heating media - two ovens or liquid baths; one set at $100 \pm 2^{\circ}\text{C}$ and the other set at $150 \pm 2^{\circ}\text{C}$. Alternatively, an aluminum block can be used for tests at 100°C .

Pressure vessels - air or oxygen pressure chambers conforming to ASTM D 454 or D 572, respectively. If this equipment is not available, pressure chambers can be constructed from pipe and fittings equipped with 1000 kPa (150 lbf/in²) pressure relief valves. It is recommended that 38-mm borosilicate glass test tubes be placed in the pressure chamber to hold the test fluid. Thus, contamination from corrosion of the pressure vessel can be avoided and each vulcanizate can be tested in a separate tube to prevent interaction effects. A suggested construction is a 125 mm (5 in) steel pipe with a plate welded to one end and a flange fitting welded to the other end. The internal height should accommodate the 38 by 300-mm tubes prescribed in ASTM D 471. This chamber can accommodate seven tubes so all vulcanizates can be tested simultaneously.

TEST MATERIALS

Liquid - ASTM Reference Coolant conforming to ASTM D 3585 supplied to each participant. Dilute this liquid with an equal volume of water.

Vulcanizates - Six production lining compounds vulcanized to the same state as linings in hose. The following vulcanizates are supplied to each participant:

1. Three green standard sheets labeled A
2. Three green standard sheets labeled B
3. Five black small sheets labeled 07016
4. Five black small sheets labeled 07040
5. Three black standard sheets labeled 611
6. Three black standard sheets labeled 615

Specimens - Cut five Type C dumbbells from each standard sheet and three Type C dumbbells from each small sheet. Allocate at random the five specimens from the standard sheets as follows: one each for (1) original properties, (2) immersion 166 h and 100°C , (3) immersion 332 h at 100°C , (4) immersion 166 h at 150°C , and (5) immersion 332 h at 150°C . Randomize the 15 specimens from the five small sheets of each vulcanizate and allocate 3 specimens for each condition. Place small holes in one end of dumbbells to be immersed in liquid. Identify dumbbells in a manner which is not affected by the liquid at high temperature and which does not affect the vulcanizate properties.

PROCEDURE

Change in mass - Determine the mass of each specimen before and after immersion in accordance with ASTM D 471, using dumbbells instead of the rectangular specimens. Calculate the change in mass of the original values.

Change in tensile strength and ultimate elongation - Determine these properties on the set of specimens reserved for original properties and on each set after being immersed in liquid in accordance with ASTM D 471 and D 412. Calculate the change in these properties as a percent of the original values.

Immersion at 100°C - Test specimens in accordance with ASTM D 471. Start test of specimens to be immersed 166 h one week after the 332 h specimens so that all specimens can be tested together for tensile properties. Immerse the 3 specimens of the same vulcanizate in a single tube containing 150 cm³ of diluted ASTM Reference Coolant. After the immersion period, examine the condition of the liquid and the specimens. Determine the mass and tensile properties of the specimens.

Immersion at 150°C - When the liquid is placed directly into the pressure chamber, use a separate chamber for each vulcanizate. Leave an air space equal to at least 1/3 the volume of the chamber. When the liquid is placed in glass tubes in the chamber, place the 3 specimens of the same vulcanizate in a single tube containing 150 cm³ of diluted ASTM Reference Coolant. To facilitate heat transfer, add about 100 cm³ of liquid to the pressure vessel outside the glass tubes. After the specimens are immersed, seal the chamber tightly to prevent leaks and place it in an oven or bath at 150°C. If possible, start test of specimens to be immersed 166 h one week after the 332-h specimens are started so that all specimens can be tested for tensile properties together. At the end of the heating period, cool the pressure vessel in air to below 40°C and open it carefully to prevent the liquid in the tubes from vaporizing explosively. Examine the condition of the liquid and the specimens. Determine the mass and tensile properties of the specimens.

Report - Record the data on the enclosed forms and send them to:

Robert D. Stiehler
Room B348 Building 226
National Bureau of Standards
Washington, D.C. 20234

APPENDIX II

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AMERICAN SOCIETY FOR TESTING AND MATERIALS

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Standard Specification for RUBBER HOSE USED IN SOLAR ENERGY SYSTEMS¹

This standard is issued under the fixed designation D 3952; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This specification establishes quality requirements for rubber hose used to convey liquids in solar energy systems. It provides producers, distributors, and users with criteria to evaluate this product. Particular applications may necessitate other requirements that would take precedence over these requirements when specified.

1.2 Annex A1 covers connections of rubber hose to the solar energy system.

1.3 This specification does not include requirements pertaining to the fabrication or installation of the hose.

1.4 This specification does not cover hose made from plastics.

2. Applicable Documents

2.1 *ASTM Standards:*

D 380 Testing Rubber Hose²

D 395 Tests for Rubber Property—Compression Set³

D 1349 Recommended Practice for Rubber—Standard Temperatures and Atmospheres for Testing and Conditioning⁴

D 1566 Definitions of Terms Relating to Rubber⁴

D 3832 Specification for Rubber Seals Contacting Liquids in Solar Energy Systems²

2.2 *Other Standards:*

ISO 1307 Rubber Hose—Bore Sizes, Test Pressures and Tolerances on Length⁵

3. Classification

3.1 *Types:*

3.1.1 *Type C*, intended for use in cold climates (below -10°C in winter).

3.1.2 *Type W*, intended for use in warm climates (above -10°C in winter).

3.2 *Classes:*

3.2.1 *Class A*, intended for use with aqueous fluids at 100°C or less.

3.2.2 *Class AT*, intended for use with aqueous fluids above 100°C .

3.2.3 *Class N*, intended for use with non-aqueous fluids.

NOTE 1—Aqueous fluids include water and anti-freeze solutions.

4. Definitions

4.1 *nipple*—a cylindrical tubular attachment, one end of which is securely inserted and retained in the end of a hose, to convey fluid to or from the hose.

4.2 Definitions of other terms are given in Definitions D 1566.

5. Ordering Information

5.1 Orders for rubber hose under this specification shall include the following information:

5.1.1 ASTM designation and date of issue,

5.1.2 Inside diameter,

5.1.3 Length,

5.1.4 Type and class (see 3.1 and 3.2),

5.1.5 Type of fluid to be conveyed,

5.1.6 Maximum service temperature,

NOTE 2—This temperature normally occurs under stagnation conditions and maximum radiation flux.

¹ This specification is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.31 on Rubber Hose.

Current edition approved Aug. 29, 1980. Published October 1980.

² *Annual Book of ASTM Standards*, Part 38.

³ *Annual Book of ASTM Standards*, Part 37.

⁴ *Annual Book of ASTM Standards*, Parts 37 and 38.

⁵ Available from American National Standards Institute, 1430 Broadway, New York, N. Y. 10018.



5.1.7 Maximum working pressure, and

5.1.8 Other requirements.

6. Standard Sizes:

6.1 The following internal diameters are standard:

mm	in.
10.0	0.38
12.5	0.50
16.0	0.63
20.0	0.75
25.0	1.00
31.5	1.25
40.0	1.50
50.0	2.00

NOTE 3—The metric and inch sizes are not identical. The metric sizes are based on the R10 series of preferred numbers given in ISO 1307.

6.2 The internal diameter shall not exceed the specified value in 6.1 and shall not be less than this value by more than 1.5 mm or 0.06 in. for sizes of 20 mm or 0.75 in. or less, or 3.0 mm or 0.12 in. for sizes above 20 mm.

6.3 The tolerance on length shall be ± 3 mm or ± 1 % of the length, whichever is larger.

7. Requirements

7.1 *Construction*—The hose shall consist of an inner tube, reinforcement, and outer cover. The outer cover may be rubber or a fibrous material (metallic or nonmetallic) that is part of the reinforcement. The inner tube and rubber cover shall be smooth, uniform in thickness, and free of pitting, pinholes, blisters, and other defects.

NOTE 4—Intentional pricks in the rubber cover to allow vapors to escape from reinforcement or lengthwise ribs on the outer cover are not considered defects.

7.2 *Inner Tube and Outer Rubber Cover*—These rubber components shall comply with the requirements given in Table 1.

7.3 *Hose*—The composite hose shall comply with the requirements given in Table 2.

7.4 *Reinforcement Material*—The reinforcement shall be stable in the presence of vapors of the heat-transfer fluid at service temperature as determined from the change in the bursting pressure of the hose in a prequalification test (see 9.3.2). The bursting pressure should decrease less than 50 % for stable material.

8. Sampling

8.1 The inspection and test procedures spec-

ified are to be used to determine conformance of products to the requirements of this specification. Each producer or distributor whose products are represented as conforming to this specification may utilize statistically based sampling plans that are appropriate for each particular manufacturing process and product characteristic. Essential records shall be kept to document with a high degree of assurance that all requirements of this specification have been met.

8.2 In case of dispute between the purchaser and the manufacturer regarding quality, five pieces of hose shall be taken from the lot at random and those attributes in dispute shall be tested for compliance with this specification. If one piece does not conform, a second set of five pieces may be taken and tested. If two or more of the ten pieces do not conform, the lot may be rejected.

9. Test Methods

9.1 Test the hose in accordance with Methods D 380 for requirements in Table 1.

9.2 Test the hose for compression set and bursting pressure by the following procedures:

9.2.1 *Compression Set*—Use the procedure for Method B in Methods D 395, except that the specimen shall consist of a piece of hose at least 50 mm in length, and the compression plates shall have the curvature of the outside of the hose as illustrated in Fig. 1. Place a metal cylinder having an outside diameter, A , equal to the inside diameter of hose given in 6.1. Measure the outside diameter, B , of the hose with the cylinder in place. Place the specimen between the compression plates and tighten the bolts until the hose is compressed to $0.75B + 0.25A$. Place the apparatus in an oven set at the temperature given in footnote A of Table 2 for 166 ± 2 h. Remove the apparatus from the oven, loosen the clamping bolts, and after 30 min, measure the outside diameter, C , of the hose at the center of the clamping area. Calculate the compression set as follows:

$$\text{Compression set, \%} = 400 (B - C) / (B - A)$$

9.2.2 *Bursting Pressure*—Determine the bursting pressure in accordance with Methods D 380.

9.3 Prequalification tests of hose shall be made for vapor transmission rate, effect on metals and fluid, and stability of reinforcement. These tests shall be made for each rubber



composition and reinforcement material for initial qualification of hose design, and are not required for quality control of manufacture. The following procedures shall be used:

9.3.1 Vapor Transmission Rate and Effect on Metals and Fluid—Prepare metal plugs from aluminum, brass, copper, and steel, 50 mm in length, from rods having a diameter equal to the inner diameter of hose given in 6.1. Cut six hose specimens 350 mm in length. Place an aluminum plug in one end of one specimen to a depth of 25 mm and clamp. Place a brass plug in second specimen and clamp. Place a copper plug in third specimen and clamp. Place a steel plug in fourth specimen and clamp. Fill these specimens with the heat-transfer fluid used in Class N hose or water used in Class A or AT hose, to about 75 mm of open end. Insert the aluminum plug in first specimen, the brass plug in the second specimen, copper plug in the third specimen, and the steel plug in the fourth specimen, so that the distance between plugs is 300 mm, and clamp. Determine the masses of the four hose assemblies and the two specimens without fluid. Place the six hose specimens in an oven at $100 \pm 1^\circ\text{C}$ when testing Class A or AT hose, and at a standard test temperature in accordance with Recommended Practice D 1349, that is, next above the maximum service temperature, but not less than 100°C , when testing Class N hose. Determine the masses after 24 h and tighten the clamps on the specimens containing fluid. Return the specimens to the oven for six more days. Remove the specimens from the oven, allow them to cool to room temperature, and determine their masses. Deduct the average loss in mass of the two dry specimens from the average loss in mass of the four specimens containing fluid. Calculate the vapor transmission rate in $\mu\text{g}/(\text{m}\cdot\text{s})$. Disassemble the four hose assemblies and examine the condition of the fluid and plugs.

9.3.2 Stability of Reinforcement—Cut ten hose specimens approximately 350 mm in length. Insert hose nipples in the ends of five specimens and clamp. Connect one nipple of each assembly to a fluid reservoir of about 100-cm³ capacity. Fill the hose and reservoir

through the other nipple with the heat-transfer fluid used in Class N hose or water in tests of Class A or AT hose to about 90 % of capacity. Close the filling nipple by any suitable means and place the five hose assemblies in an oven at $100 \pm 2^\circ\text{C}$ when testing Class A or Class AT hose, and at a standard test temperature in accordance with Recommended Practice D 1349, that is, nearest the maximum service temperature, but not less than 100°C , when testing Class N hose. Examine the fluid level in the assemblies after 2 and 4 months, and add fluid if the level has decreased below 80 % of capacity. After 6 months, remove the specimens from the oven, disconnect the reservoirs, and measure the bursting pressure of each specimen, including the five specimens that were not filled and heated with fluid. Calculate the change in the average bursting pressure of the two sets of specimens as a percent of the bursting pressure of specimens not heated.

10. Marking

10.1 The following information shall be marked on the rubber hose:

10.1.1 Name, brand, or trademark of the manufacturer,

10.1.2 Type and class,

10.1.3 Reference to this specification, ASTM D 3952, and

10.1.4 Other information required by the manufacturer or the purchaser.

11. Packaging

11.1 Hose shall be protected by suitable packaging to prevent damage during shipment or storage prior to installation in the solar energy system.

12. Instructions

12.1 Installation instructions should be included with each package. Annex A1 gives guidance on information to be included to assure long leak-free service. Instructions are not necessary for hose supplied with end fittings unless special precautions during installation or maintenance procedures must be followed.



TABLE 1 Requirements for Inner Tube and Outer Rubber Cover

Property	Requirement
Ultimate elongation, min, %	250
Tensile strength, min, MPa	6.2
psi	900
Resistance to heating: ^a	
Change in ultimate elongation, max, %	-40
Change in tensile strength, max, %	-20
Resistance to heat transfer fluid: ^b	
Change in ultimate elongation, max, %	±25
Change in tensile strength, max, %	±25
Change in hardness, max	±10
Change in volume, max, %	±15
Resistance to ozone of outer cover, 100 mPa, ^c for 166 h at 40°C	no cracking
Resistance to low temperature, Type C hose only, -40°C	no cracking

^a Class A hose shall be heated at 125 ± 2°C for 166 ± 2 h. Class AT and N hose shall be heated for 166 ± 2 h at a standard temperature in accordance with Recommended Practice D 1349, that is, between 25 and 49°C above the maximum service temperature, but not less than 125°C. These test temperatures are: 125, 150, 175, 200, 225, and 250°C.

^b The inner tube of Class A and AT hose shall be immersed in a mixture containing equal volumes of ethylene glycol and water for 166 ± 2 h at 100 ± 2°C. The inner tube of Class N hose shall be immersed in heat transfer fluid used in the solar energy system for 166 ± 2 h at a standard test temperature in accordance with Recommended Practice D 1349 next above the maximum service temperature, but not less than 100°C. If the vapor pressure of the fluid is above atmospheric pressure at the test temperature, the next lower standard test temperature shall be used. (A method is being considered in Subcommittee D11.31 that would subject the inner tube of the hose to volatile fluids above their boiling point in pressure vessels so that the inner tube can be tested above the maximum service temperatures regardless of fluids used.)

^c 100 mPa ozone partial pressure is equivalent to 100 ppm at standard atmospheric pressure of 100 kPa.

TABLE 2 Requirement for Composite Hose

Property	Requirement
Compression set, max, %	85 ^a
Bursting pressure, min	five times working pressure
Vapor transmission rate, max, μg/(m·s)	3.5 ^{b,c}
Effect on aluminum, brass, copper, and steel	no corrosion ^d
Effect on fluid	no sediment or dark discoloration ^e
Stability of reinforcement, maximum decrease in bursting pressure, %	50

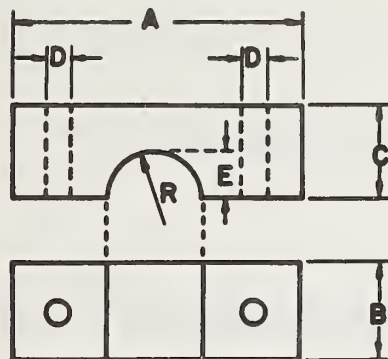
^a Compression set is measured after conditioning under 25 % compression for 166 ± 2 h at 100 ± 2°C for Class A hose and at the standard test temperature in accordance with Recommended Practice D 1349, that is, between 25 and 49°C above the maximum service temperature, but not less than 125°C. These test temperatures are: 125, 150, 175, 200, 225, and 250°C.

^b Equivalent to 0.9 g/day for a specimen having an internal length of 300 mm between plugs.

^c The requirement for Class N hose with specified fluids should be agreed upon between the manufacturer and the purchaser of the hose.

^d Condition of the plugs at the end of the test for vapor transmission rate. Discoloration of the plugs without corrosion is permitted.

^e Condition of the fluid at the end of test for vapor transmission rate. Slight cloudiness or slight discoloration is permitted.



- A - 5R, MINIMUM
- B - 25 MM
- C - 10 MM + R, MINIMUM
- D - HOLES TO CLEAR BOLTS
- E - 3/8 O.D. + 1/8 I.D. OF HOSE, MAXIMUM
- R - 1/2 Q.D. OF HOSE, MINIMUM

FIG. 1 Compression Clamp (Two Required)

ANNEX

A1. CONNECTION OF HOSE TO SOLAR ENERGY SYSTEM

A1.1 The means for fastening the hose to components in the solar energy system is critical. The two mechanisms commonly employed are through end fittings on the hose or clamps. Materials that resist corrosion are essential. The following provisions are general requirements and not complete specifications for these connections.

A1.1.1 *Hose Nipples*—The outside diameter shall be not less than the specified value in 6.1 and shall not exceed this value by more than 3 mm, including serrations or enlargement to prevent slippage.

A1.1.2 *End Fittings*—Hose equipped with end fittings shall withstand a minimum tensile force of 1.3 kN (300 lbf). In the bursting pressure test (9.2.2), the hose shall not move out of the end fittings more than 1.6 mm (0.06 in.). Rubber seals in end fittings shall be in accordance with Specification D 3832. The design of the end fitting shall be agreed upon between the purchaser and the manufacturer.

A1.1.3 *Clamps*—Hose intended for use with

clamps shall be shipped with instructions for proper clamping to prevent slippage of hose from the connecting tubes in the solar energy system and cutting of the hose by the clamps. The instructions should emphasize that one or more ridges between the clamp and the end of the hose nipple is essential to prevent slippage, and that the clamps should have a wide surface to support the clamping forces without cutting of the hose. The instructions should also include recommendations for use of adhesives to prevent leakage and reduce or eliminate maintenance during long service.

A1.1.4 *Compatibility*—The rubber hose, metal components, and circulating fluid should be compatible in order to minimize metal corrosion and deterioration of rubber hose and fluid. Specifications for compatibility beyond the provisions in Table 2 and 9.3 should be agreed upon between the purchaser and the manufacturer.

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11. ABSTRACT <i>(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)</i> Class AT hose in ASTM Standard D 3952-80, Specification for rubber hose used in solar energy systems, is specified for use with aqueous liquids above 100°C. The lining of this hose is subjected to immersion tests at 100°C. The purpose of this study is to determine whether immersion tests in aqueous liquids above maximum service temperature are necessary in the hose standard. The results of an interlaboratory test indicate that Class AT hose should be subjected to immersion tests above maximum service temperature. By inference, Class N hose used with a volatile heat transfer liquid at a temperature above its boiling point should be similarly tested above maximum service temperature. The study also indicates that ASTM Reference Coolant (ethylene glycol base) is not stable at 150°C. In addition to the hose lining, the stability of the heat transfer liquid above maximum service temperature must be determined to assure satisfactory performance of the system. A proposed revision of ASTM D 3952 is included in the report. It provides for immersion tests above maximum service temperature of Class AT and Class N hose linings used with heat transfer liquids above their boiling points.			
12. KEY WORDS <i>(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)</i> hose, hose specification, hose immersion test, heat transfer liquid, glycol antifreeze stability, rubber hose, solar energy systems.			
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